PATENT SPECIFICATION

DRAWINGS ATTACHED

Inventors: THOMAS HENRY BULL and ARTHUR MURRAY GARDINER KINNEAR

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COMPLETE SPECIFICATION

Production of Oriented Polyester Filaments and of Staple Fibres Therefrom

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, of Imperial Chemical House, Millbank, London, S.W.1., a British Company. do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for the manufacture of staple fibres from oriented polyester filaments.

Melt spun polyester filaments having a low birefringence after melt spinning are subjected to a drawing operation in which molecular orientation is brought about when the filaments are elongated 2—5 times their length with a desirable increase in birefringence and an improvement in textile properties.

We have found that heavy undrawn tows above 100,000 denier can be drawn satisfactorily at desirable high commercial draw speed i.e. the speed of the tow on leaving the last draw roll from 100—600 feet per minute to acceptable defect levels. Moreover we have found that this high speed drawing may be carried out with a single draw frame bath at comparatively low temperatures and the use of steam and liquid sprays which were regarded to be essential on a commercial scale are not required in our process.

The polyester filaments which can be meltspun and drawn according to our invention are made from polymers based on terephthalic acid, the best known representative being polyethylene terephthalate, although we believe that the process is also applicable to other fibre forming polyesters and copolyesters based on terephthalic acid. Our process is particularly suitable for the drawing of polyester filaments having an intrinsic viscosity below 0.49.

According to our invention we provide a process for melt-spinning and orienting polyester filaments by drawing up to 5 times their spun length in the form of a tow, comprising submitting filaments issuing from a spinneret, to a quenching gas stream at a distance 0 to 10 cm. from the spinneret followed by wetting the tow which in the form of a flattened band has a linear density of at least 100,000 denier per inch at feed speeds from 100—600 feet per minute in a pre draw frame bath substantially at room temperature, and straightening any loops in the tow and lubricating the tow, followed by tensioning the tow, e.g. by passage over and under and in contact with alternate fixed tensioning bars in a sinuous path, then drawing the tow by forwarding it to feed rolls, the last of which is at partially immersed in a draw frame bath filled with aqueous liquid, maintained at a temperature within the range 50—100°C, then through the aqueous liquid to draw rolls at least one of which, preferably the first, is partially immersed in the draw frame bath and rotating at a linear speed at least twice that of the feed rolls to draw the filaments in the draw-frame bath at a draw ratio ranging from 2—5 and to give the required orientation and draw ratio. The temperature of the liquid, preferably water, in the draw frame bath is 50°C to 100°C. Our process has the advantage that total immersion of the tow in the draw zone ensures that surface tension which tend to cause adjacent filaments to adhere or couple are minimised, and secondly that draw ratios above 1:2 can be used whilst maintaining the draw zone on the immersed surface of the feed roll. This is believed to become possible because of the combination of temperature and tension gradients created in the

draw bath in conjunction with the tensioning of the tow before the feed rolls and complete immersion in the draw bath. The movement of the draw zone is restricted by both the temperature and tension gradients on the partially immersed feed roll, and hence permits a wider range of draw ratios than can be attained using a simple temperature gradient.

Our process is particularly suitable for the meltspinning and drawing of tows from polyester filaments having a low intrinsic viscosity below 0.49. Very satisfactory results have been obtained with polyethylene terephthalate of intrinsic viscosities of 0.38—47. By room temperature we mean temperatures of about 10°—25°C. Fibres with a birefringence of $180—220\times10^{-3}$ are desirable. The filaments in the tow after melt spinning and quenching but before drawing should have a low birefringence of 0 to 10×10^{-3}.

The established techniques for the melt spinning and drawing of fibres of high intrinsic viscosity require modification when applied to polymer or tow of reduced intrinsic viscosity, particularly in respect of the maintenance of a stable threadline during melt spinning, and the achievement of successful drawing of the filaments particularly in the form of heavy tows, prior to crimping and cutting into staple fibres.

Moreover, we have found that staple fibres having an intrinsic viscosity above the range of 0.28—0.45 disclosed in Specification 840,796, have other advantages such as better abrasion resistance and tenacity.

We therefore also provide a process for making staple fibres which in fabric form have a reduced tendency to pilling comprising melt spinning polyethylene terephthalate of an intrinsic viscosity between 0.38 and 0.49, particularly between 0.45 and 0.48, submitting the filaments issuing from the spinneret to quenching by a gas at a distance 0 to 10 cm. from the spinneret followed by drawing the filaments in a liquid medium preferably water or a dilute aqueous solution or suspension as described. Air at 15°C to 40°C is suitable for quenching. Drawing of 2 to 5 times, preferably 3.5 to 4.5 times, the undrawn length of the "as spun" filaments, is suitable. A temperature of 50°C to 90°C preferably 75° to 80°C is suitable for a liquid medium in which the filaments are drawn.

The fibre intrinsic viscosity necessary to avoid unsightly pills is dependent, amongst other things, on fabric structure. In open structures, such as those found in knitted garments, an intrinsic viscosity as low as 0.38 may be necesary.

After drawing the filaments may be crimped and the crimp heat set so that the residual shrinkage in boiling water for one minute is less than 1%. Because of the conditions of spinning and drawing there are no coalesced or partly drawn length of filaments or fibres.

The intrinsic viscosity of our staple fibres is preferably between 0.44 to 0.46 because in this narrow range fibres having the required high tenacity and low extensibility can be obtained by drawing under our preferred conditions.

If the intrinsic viscosity of the polyester is below the specified limits, fibres with a lower tenacity are obtained and difficulties associated with melt spinning are increased. Fibres with a tenacity of at least 3 grams per denier are easily obtained by our process.

If the intrinsic viscosity is increased above the specified limit, the tendency to pilling of fabrics containing the staple fibres is increased to an extent which renders them unsuitable for many otherwise useful fabric constructions. Highly twisted yarns, singeing of the yarns and fabrics, and tighter fabric constructions, however, reduce pilling.

We also find that the second order transition temperature of 100°C—110°C of the polyester fibres when wet can be related to the pleat retention and wrinkle resistance performance during washing of fabrics made from such low intrinsic viscosity fibres, which are very desirable for apparel uses and polyesters having a second order transition temperature within this range are therefore preferred. It is preferred to use spinneret holes during melt spinning which are larger than usual for the required textile deniers, in the drawn condition of about 1 to 6 denier.

Circular orifices having a diameter greater than 15/1000″ up to 30/1000″ may be used. Instead of circular orifices, non-circular orifices may be used, which, however, have an orifice-area corresponding to the area of the preferred circular large orifices.

The drawing (accompanying the provisional specification) illustrates the drawing apparatus which may be used in the process of our invention. A pre-drawframe bath (1) is filled with water or an aqueous solution containing processing aids. Guide rolls (2) (3) & (4) are used to guide a heavy tow into the bath. Above the guide roll (4) there is a set of stationary tensioning bars (5) over and under which the tow coming from the immersed roll (4) is passed to suitably positioned draw rolls (6) (7) (8) (9) (10) (11) & (12) which are each driven at a uniform speed and rolls (6) & (7) are in addition provided with resilient free running nip rolls (shown hatched). Feed roll (12) is partly immersed in a draw frame bath (13) and is provided with heating means and thermal regulating means (not shown) at the other end of the drawframe bath (13), also partly immersed is a draw roll (14) and additional draw rolls (15) (16) (17) (18) (19) & (20), outside the draw frame bath and roll (15) is provided with a resilient nip roll which is driven by surface contact from the tow by

roll (15). The draw rolls are driven at predetermined faster uniform speeds than the feed rolls. The speed of the tow on leaving the last draw roll is herein referred to as the draw speed.

The following Examples illustrate but do not limit our invention.

Example I

Polyethylene terephthalate of intrinsic viscosity 0.67 was melt spun into multi-filament yarns of 10.6 denier per filament, the birefringence of the undrawn yarn being 3.1×10^{-3}. Spinning was carried out from a spinneret consisting of 250 circular holes of 0.009″ diameter. The filaments coming from the spinneret were submitted to a quenching air stream directed radially outwards from the axis of the cone formed by the issuing filaments, at a distance 3″ from the spinneret face and extending downwards for 10″. The temperature of the air entering the air duct was 20°C. The spun yarns were plied up to give a 260,000 denier tow consisting of 25,000 filaments which was spread over 2″ and drawn, by passage through a pre-draw frame bath (1), by passage over guide rolls (2, 3 and 4) so that the tow became completely immersed in the pre-draw frame bath containing water at 20°C.

From the guide roll (4) the tow is tensioned by passage over and under tensioning bars (5) and then to feed rolls (6, 7, 8, 9, 10, 11 and 12) rotating at a uniform linear speed of 66 feet per nimute, the last feed roll (12) is partly immersed in a draw frame bath (13), filled with water maintained at 80°C. Also partly immersed in the draw frame bath is the first of a set of draw rolls (14) rotating at a linear speed of 300 feet per minute under which the tow is passed and then over draw rolls (15—20) rotating with the same speed as partially immersed draw roll (14). Rolls 6, 7 and 15 are provided with nip rolls. The drawn tow may be cut into staple lengths or wound up. Before cutting, the tows and filaments may be crimped and the crimp heat-set by treatment with steam at about 140°C for half an hour or by setting using dry heat at about 135°C for 15 minutes. The drawn filaments or fibres are uniformly oriented and the defect level is commercially acceptable, there being less than 5 p.p. 100,000 of undrawn fibres as determined by dye-fleck test.

Example II

Polyethylene terephthalate of I.V. 0.47 was melt spun into multi-filament yarns of 10.6 d.p.f., and of birefringence 2.36×10^{-3}. Spinning was carried out from a spinneret comprising 250 holes of cruciform section, the arms of the holes being .015″×.004″. The threadline was quenched by a flow of air directed radially outwards from the axis of the threadline cone, the quench being applied at a level of 2″ below the spinneret face and extending downwards for 8″. The flow of air was 50 cu.ft. per minute. The spun yarn was plied to give a 260,000 denier tow which was passed through a pre draw frame bath containing a 3% aqueous solution of antistatic agent "Zelec" NE (Registered Trade Mark), believed to be a diethanolamine salt of an acid phosphate ester of long chain (C_8—C_{10}) fatty alcohols, at room temperature, and thereafter through the apparatus as in Example I and delineated in Figure 1. The drawbath contained the same solution maintained at 78°C. The feed rolls rotated at a linear speed of 55 f.p.m. and the draw rollers at 250 f.p.m. The drawn tow contained less than 5 p.p. 10^5 of undrawn fibres, as determined by the dye fleck test.

Example III

A copolymer of polyethylene terephthalate containing 6 moles. % sebacic acid residues and of I.V. 0.47 was melt spun into multifilament yarn of 10.6 d.f.p, and birefringence 1.98×10^{-3}, using the spinneret and quench arrangements described in Example II. The spun yarn was plied to give a 260,000 denier tow which was drawn on the apparatus described in Example I the pre draw frame bath containing a 3% aqueous solution of "Zelec NE" at room temperature and the drawbath containing the same solution at 70°C. The feed rolls rotated at a linear speed of 55 f.p.m. and the draw rolls at 250 f.p.m. The drawn tow contained less than 5 p.p. 10^5 of undrawn fibres as determined by the dyefleck test.

Example IV

Polyethylene terephthalate of I.V. 0.67 was melt spun into multi-filament yarns of 4.8 d.p.f. and of birefringence 5.17×10^{-3}. The spinneret used comprised 508 circular holes of 0.009″ diameter. A radial air flow quench was applied as in Example 2 but in this case at a level of $\frac{3}{8}$″ below the spinneret face, and at a flow rate of 40 cu. ft./min. The yarn was plied to 250,000 denier tow which drawn on the apparatus described, both baths containing water, the pre draw bath being at room temperature and the drawbath at 78°C. The feed rolls rotated at a linear speed of 122 f.p.m. and the draw rolls at 500 f.p.m. The drawn tow contained less than 5 p.p. 10^5 of undrawn fibres as determined by the dyefleck test.

In the examples, the width of the tow was adjusted to spread to 2″ between spacing rollers so that the linear density of the tow was 130,000 denier per inch in Example I—III and 125,000 denier per inch in Example IV.

Fabrics of a 2×2 twill construction made from 3 denier per filament 4″ length fibres when melt spun and drawn and having an

intrinsic viscosity of 0.45—0.48 and when spun on the worsted system into 2/28's count yarns, show no pills after brushing and sponging for 200 minutes as described in A.S.T.M. 1958 D 1375—55T, Page 515.

The aqueous liquid in the draw frame baths may contain a solution or suspension of a surface active agent as well as other agents for imparting antistatic or other properties and lubrication to the polyester filaments, if desired.

For the purposes of comparison when the liquid in the draw frame bath is replaced by water sprays or by steam sprays drawing conditions of the heavy tows of intrinsic viscosity less than 0.49 become more uneven as evidenced by an increase in the defect level which under the most favourable conditions is above 50 p.p. 100.000.

Similarly, if the pre draw frame bath or the tensioning baths are removed the defect level is increased beyond commercially acceptable limits. This was particularly enhanced when drawing polyethylene terephthalate tows of an intrinsic viscosity of 0.49 and below.

When the air quench of the filaments below the spinneret is stoped by shutting off the air supply, unsteady spinning conditions associated with a phenomenon called kneeing results; during the subsequent drawing process many filament breaks occur and the defect level rises above commercially acceptable limits, when the intrinsic viscosity is below 0.49.

Suitable dwell times for the quenched filaments, after lubrication in the pre draw frame bath and after tensioning between the feed and draw rolls in the draw frame bath filled with aqueous liquid are 0—2 seconds to 3.7 seconds; draw-bath lengths of at least 24 inches at tow speeds of 600 ft./min. and 75 inches at tow speeds of 100 ft./minute may be used.

WHAT WE CLAIM IS:—

1. A process for melt-spinning and orienting polyester filaments by drawing up to 5 times their spun length in the form of a tow, comprising submitting filaments issuing from a spinneret to a quenching gas stream at a distance 0 to 10 cm. from the spinneret, followed by wetting the tow which in the form of a flattened band has a linear density per unit width in the drawn state of at least 100,000 denier per inch at draw speeds on leaving draw rolls of from 100—600 feet per minute in a pre draw frame bath substantially at room temperature, and straightening any loops in the tow and lubricating the tow, followed by tensioning the tow, e.g. by passage over and under and in contact with alternate fixed tensioning bars in a sinuous path, then drawing the tow by forwarding it to feed rolls, the last of which is at least partially immersed in a draw frame bath filled with aqueous liquid, maintained at a temperature within the range 50—100° C, then through the aqueous liquid to draw rolls at least one of which, preferably the first, is partially immersed in the draw frame bath and rotating at a linear speed at least twice that of the feed rolls to draw the filaments in the drawframe bath at a draw ratio ranging from 2—5 and to give the specified orientation and draw ratio.

2. A process according to Claim 1 in which the temperature of the quenching gas stream is 15°C—40°C.

3. A process according to Claim 1 or Claim 2 in which the temperature of the liquid in the pre draw frame bath is 10—25°C and that in the draw frame bath 75°C to 80°C.

4. A process according to any one of the preceding claims in which the draw zone is maintained on the immersed surface of the last feed roll.

5. A process according to any one of the preceding claims in which the polyester filaments have an intrinsic viscosity below 0.49.

6. A process according to any one of the preceding claims in which the polyester filaments are made from polyethylene terephthalate and have an intrinsic viscosity of 0.38—0.47.

7. A process according to any one of the preceding claims, in which during melt spinning spinneret holes are used which are larger than usual for the required textile deniers, in the drawn condition of 1—6 denier.

8. A process according to Claim 7 in which during melt spinning circular spinneret orifices are used with a diameter of 15 thousands up to 30 thousands of an inch.

9. A process according to Claim 7 in which during melt spinning, non circular spinneret orifices are used.

10. A process substantially as described with particular reference to the examples 1—5.

11. An apparatus for use in the process of any one of the preceding claims, substantially as described with particular reference to the drawing.

12. Melt spun and drawn polyester filaments and fibres having a birefringence of $180—220 \times 10^{-3}$ whenever made by a process according to any one of the preceding claims, which in the form of fabrics of a 2×2 twill construction from 2/28's count yarns show no pills after brushing and sponging for 200 minutes, as described.

WALTER SCOTT
Agent for the Applicants.

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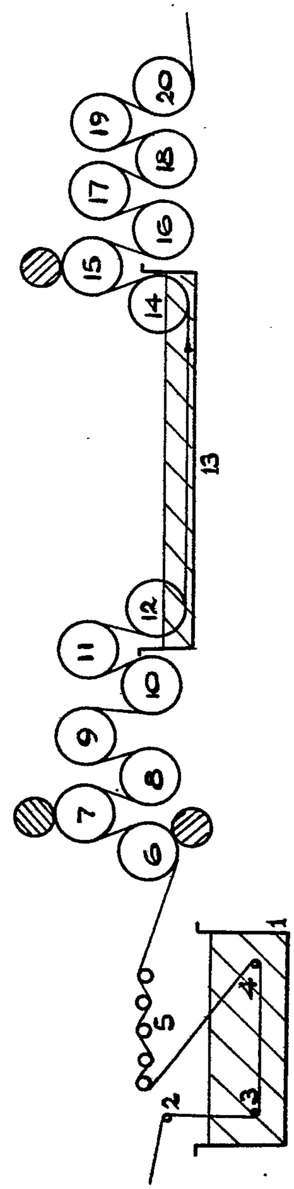
992670 PROVISIONAL SPECIFICATION

1 SHEET

This drawing is a reproduction of the Original on a reduced scale



DERWENT-ACC-NO: 1968-25275P

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TITLE: Prodn. of oriented polyester filaments

PATENT-ASSIGNEE: IMPERIAL CHEM IND LTD[ICIL]

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE
GB 992670 A	May 19, 1965	EN
CA 748355 A	July 15, 1974	EN
DE 1435495 A		DE
US 3259681 A		EN
NL 142735 B		NL

APPLICATION-DATA:

PUB-NO	APPL-DESCRIPTOR	APPL-NO	APPL-DATE
DE 1435495A	N/A	1963DE-I023596	April 24, 1963

INT-CL-CURRENT:

TYPE	IPC DATE
CIPS	D01F6/62 20060101
CIPS	D02J1/22 20060101

ABSTRACTED-PUB-NO: GB 992670 A

BASIC-ABSTRACT:

Melt-spinning and orienting polyester filaments by drawing up to five times their spun length in the form of a tow, comprises submitting filaments issuing from a spinneret to a quenching gas stream at a distance 0 to 10 cm. from the spinneret, followed by wetting the tow which in the form of a flattened band has a linear density per unit width in the drawn state of at least 100,000 denier per inch at draw speeds on leaving draw rolls of from 100-600 feet per minute in a pre-draw frame bath substantially at room temperature, and straightening any loops in the tow and lubricating the tow, followed by tensioning the tow.

TITLE-TERMS: PRODUCE ORIENT POLYESTER FILAMENT

DERWENT-CLASS: A32 F01